DELTAMETHRIN (135)

EXPLANATION

Deltamethrin was evaluated in 1980 and in most years since, most recently in 1990. MRLs for unprocessed wheat bran, wheat flour and wheat wholemeal are held at Step 7B pending JMPR review. At the 20th (1988) Session of the CCPR it was suggested (ALINORM 89/24, para 141) that the proposed MRL (2 mg/kg) for unprocessed wheat bran was too low, and that at least 3 mg/kg was necessary. The manufacturer would provide new data on this aspect. Because of the relation between wheat bran, flour and wholemeal, the CCPR decided that MRLs for all three commodities should be held at step 7B to await the JMPR's review. At the 24th (1992) Session of the CCPR the manufacturer and the delegation of Australia informed the Committee that data had been supplied for review by the 1992 JMPR.

The 1990 JMPR evaluated numerous supervised trials where deltamethrin was used for veterinary treatments of cattle, calves, sheep and pigs as dips, pour-ons and sprays. At the 24th (1992) Session of the CCPR it was suggested (ALINORM 93/24, para 140) that a longer waiting period before slaughter would assist in attaining the lowest possible residue. (Some countries have a nil waiting period). However, it was noted that in some circumstances involving quarantine requirements it was necessary to have a short waiting period. The Committee decided to advance the MRL for meat to Step 8. Further information on deltamethrin residues in cattle was made available to the present Meeting.

Information on horticultural and agricultural uses in Spain and residue data on artichokes and tamarillos were also made available to the Meeting.

USE PATTERN

Deltamethrin is used as a post-harvest insecticide on stored cereals to control insect storage pests. It is normally used at 0.5-1 g ai/t when used alone, or at 0.25-0.5 g ai/t when used in combination with piperonyl butoxide as a synergist. Registered uses of deltamethrin as a post-harvest insecticide are listed in Table 1. Registered formulations include dusts (0.5, 1 or 2 g ai/kg), emulsifiable concentrates (25 g ai/l with 250 g piperonyl butoxide/l) and an ultra low-volume concentrate (6 g ai/l with 60 g piperonyl butoxide/l).

In Spain deltamethrin is registered for field application on vegetables at 0.75-1.25 kg ai/ha for the control of Colorado beetle, aphids, whitefly, thrips and caterpillars. Horticultural and agricultural uses in Spain are summarized in Table 2.

In New Zealand deltamethrin is used on tamarillos for whitefly control at spray concentrations of 2 g ai/hl before flowering, and 0.62-1.25 g ai/hl after flowering, with a PHI of 60 days.

Table 1.post-harvest uses of deltamethrin

Commodity	Country	Application rate, g ai/t	Withholding period
Barley	Czechoslovakia	0.5	42 days
Beans	France	0.5-1	2 days
Cereals	Algeria	0.6	
	China	0.5-1	
	Cuba	0.5-0.7	
	France	0.5-1	2 days
	Guatemala	0.5-1	
	Hungary	0.5	14 days
	Italy	0.5-1	42 days
	Mexico	0.5-1	
	Morocco	0.5	
	Philippines	0.5-1	
	Romania	0.5	
	Spain	0.5-1	
	Tunisia	0.5-1	
	Venezuela		
	Yugoslavia	0.25-0.5	56 days
Coffee	Brazil	0.35-0.5	15 days
Maize	Argentina	0.25-0.5	
	Czechoslovakia	0.5	42 days
Millet	Brazil	0.35-0.5	30 days
Oats	South Africa		
Potatoes	Algeria	0.75	
	Tunisia	0.75	
Pulses	Algeria	0.6	
	Argentina	0.25-0.5	
	Spain	0.5-1	
Rice	Argentina	0.25-0.5	
	Brazil	0.35-0.5	15 days
Rye	South Africa		
Sunflower	Hungary	0.5	14 days
Wheat	Argentina	0.25-0.5	
	Brazil	0.35-0.5	30 days
	Czechoslovakia	0.5	42 days
	South Africa		

Table 2. Registered field and glasshouse uses of deltamethrin(25 g ai/l EC) on horticultural and agricultural crops in Spain

CROP	Application 1/		PHI, days
	Rat e per application kg ai/ha	Spray conc kg ai/hl	
Cereals	0.0075-0.0125 f	0.00075-0.00125	3
Citrus fruits	0.0075-0.0125 f	0.00075-0.00125	3
Grapes	0.0075-0.0125 f	0.00075-0.00125	3
Lucerne	0.0075-0.0125 f	0.00075-0.00125	3
Maize	0.0075-0.0125 f	0.00075-0.00125	3
Olive	0.0025 f	0.0125	3
Pome fruits	0.0075-0.0125 f	0.00075-0.00125	3
Potatoes	0.0075-0.0125 f g	0.00075-0.00125	3
Stone fruits	0.0075-0.0125 f	0.00075-0.00125	3
Vegetables	0.0075-0.0125 f g	0.00075-0.00125	

1/ f: field use. g: glasshouse use.

National authorised veterinary uses of deltamethrin were reported in 1990 (Evaluations, 117).

In Australia deltamethrin is registered for use in controlling lice and flies on cattle. A pour-on formulation is applied as a single stripe along the backline at 0.05-0.1 g ai per 100 kg animal live-weight. This means it is used at 0.05-0.1 g ai per animal for animals up to 100kg, 0.1- 0.2 g ai per animal for those weighing 101-200 kg, and so on. Alternatively animals are treated by hand-spraying, in a spray-race or in a plunge dip, with the spray or dip containing deltamethrin (50 mg ai/l) in conjunction with an organophosphorus compound. No withholding period before slaughter is required.

RESIDUES RESULTING FROM SUPERVISED TRIALS

Artichokes. Information was made available to the Meeting by Spain on supervised residue trials on artichokes (Table 3). The CXL for globe artichoke is 0.05 mg/kg; at an application rate of 0.26 kg ai/ha (a highly exaggerated rate) deltamethrin residues comply with the CXL 7 days after application.

Table 3. Deltamethrin residues in artichokes from supervised spray trials on field plots in France and Spain

COUNTRY, year	Application		PHI, days	Deltamethrin residues, mg/kg
	Spray conc., kg ai/hl	Spray rate, kg ai/ha		
FRANCE, 1983	0.0015	0.03	13, 27	<0.01, <0-01
	0.0015	0.03	20, 34	<0.01, <0-01
	0.0012	0.024	67, 82	<0-01, <0-01
SPAIN, 1987	0.013	0.26	0	0.16, 0.08, 0.36
			3	0.12, 0.02, 0.06
			7	0.05, 0.04, 0.05
			14	0.03, 0.03, 0.03
SPAIN, 1989	0.013	0.20	0	0.07, 0.10, 0.14
			3	0.02 , 0.04, 0.04
			7	- , 0.01, 0.01

Tamarillos. Supervised trials in New Zealand (Table 4) demonstrated that when deltamethrin was used according to GAP (0.6-1.25 g ai/hl, 60 days PHI) residues on tamarillos were close to 0.01 mg/kg.

Table 4. Deltamethrin residues in tamarillos from supervised New Zealand trials in 1987 using a 25 g/l EC formulation (Study no. HOECHS 2873)

Application			Deltamethrin residues, mg/kg, at days after final treatment							
Spray conc g ai/hl	Rate, kg ai/ha	No	0	1	3	7	14	21	27-28	56
0.625	0.0125	1	0.009			0.003	0.002		0.002	<0. 002
0.6 + oil	0.012	11	0.024	0.031	0.031	0.031		0.032	0.025	0.011
1.2	0.024	11	0.012	0.014	0.014	0.003		0.005	0.014	0.008

Milk. Application of a deltamethrin pour-on formulation to dairy cows in supervised trials in France in 1989 produced the highest mean residues in the milk on day 3 after treatment (de Laistre Banting 1989a,b). Residue data are shown in Table 5. There was a wide variation from animal to animal in residue levels, and for individual animals the highest residues were found on days 2, 3, 4, 5, and 6 after treatment.

Table 5. Deltamethrin residues in milk following application of a pour-on formulation (7.5 g/l) directly to 10 dairy cows at 1.1 mg ai/kg live-weight
(Ref 1, de Laistre Banting, 1989a) and at 0.77-0.93 mg ai/kg live-weight
(Ref 2, de Laistre Banting, 1989b) in supervised trials in France in 1989.

Interval after treatment, days 1/	Deltamethrin residues in milk, mg/kg			
	Ref 1		Ref 2	
	Median	Range	Median	Range
0 E	<0.001	<0.001-0.002	<0.001	<0.001
1 M	<0.001	<0.001-0.002	<0.001	<0.001-0.002
1 E	<0.0015	<0.001-0.004	0.002	<0.001-0.003
2 M			0.002	<0.001-0.007
2 E			0.002	<0.001-0.009
3 M	0.004	0.001-0.008	0.003	0.001-0.007
3 E	0.005	0.002-0.010	0.005	0.002-0.008
4 M			0.003	<0.001-0.007
4 E			0.004	<0.001-0.013
5 M			0.002	<0.001-0.007
5 E			0.002	<0.001-0.008
6 M			0.002	<0.001-0.005
6 E			0.002	<0.001-0.010
7 M	0.001	<0.001-0.005	0.001	<0.001-0.006
7 E	0.003	<0.001-0.007		

1/ E: evening milking. M: morning milking.

Deltamethrin residues in milk from lactating dairy cows treated with a pour-on formulation showed wide variations between animals (Table 6). Maximum residues were detected 1-2 days after treatment. The deltamethrin residues in Table 6 are reported on a whole milk basis, calculated by dividing the reported concentration in the butterfat by 25.

Table 6. Deltamethrin residues in milk following application of an aqueous suspension pour-on formulation (7.5 g/l) directly to 3 lactating dairy cows at 15 ml per 100 kg live-weight (GAP, 1.2-1.3 mg ai/kg) and to 3 others at 30 ml per 100 kg live-weight (2.3-2.6 mg ai/kg) in a supervised trial in Australia in 1991 (Shepherd *et al.*, 1991). Treatment took place after morning milking on day 0.

Interval after treatment, days 1/	Deltamethrin Residues in milk, mg/kg					
	15 ml/100 kg			30 ml/100 kg		Un-treated
-1 M E	<0.001	(6 samples)		<0.001	(6 samples)	<0.001
0 M	<0.001,	<0.00,	<0.001	<0.001,	<0.001, <0.001	<0.001
0 E	<0.001,	0.002,	0.001	<0.001,	0.001, 0.001	<0.001
1 M	0.009,	0.010,	0.004	<0.001,	0.001, 0.009	0.001
1 E	0.019,	0.020,	0.008	0.002,	0.001, 0.022	<0.001
2 M	0.016,	0.015,	0.015	0.014,	0.001, 0.020	<0.001
2 E	0.014,	0.008,	0.010	0.012,	0.001, 0.015	<0.001
3 M	0.008,	0.007,	0.006	0.011,	0.002, 0.010	<0.001
3 E	0.006,	0.006,	0.003	0.012,	0.001, 0.009	0.001
5 M,	0.002,	0.001,	0.002	0.005,	0.001, 0.003	<0.001
5 E	0.001,	0.002,	0.002	0.005,	0.001, 0.003	0.001
7 M	0.001,	0.001,	0.001	0.006,	0.002, 0.001	<0.001
7 E	0.001,	0.001,	0.001	0.004,	0.002, 0.001	<0 . 001
10 M	0.001, ,	<0.001	<0.001	0.002,	0.001, 0.001	<0.001
10 E	0.001,	0.001,	<0,001	0.002,	0.002, 0.001	<0.001

1/ E: evening milking. M: morning milking.

In a 1981 supervised dairy cow trial in Australia deltamethrin residues were measured in rendered butterfat from milk produced by animals treated with a pour-on formulation (Kieran *et al.*, 1981). The rate of application (2.1 mg ai/kg live-weight) was twice the recommended rate. The deltamethrin residues in Table 7 are reported on a whole milk basis, calculated by dividing the reported concentration in the butterfat by 25. The highest residues were found in the milkings 24 and 36 hours after treatment, with a residue decline evident on day 2.

Table 7. Deltamethrin residues in milk following application of a pour-on formulation (25 g/l, xylene-based) directly to 6 lactating dairy cows at 8.1-8.6 ml per 100 kg live-weight (2.1 mg ai/kg) in a supervised trial in Australia in 1980 (Kieran et al., 1981). Residues were measured in the rendered butterfat, and have been calculated on a whole milk basis by dividing the concentration in the butterfat by 25.

Interval after treatment	Deltamethrin residues in milk mg/kg
Pre-treatment	<0.002 (6 samples)
12 hours	0.011, 0.010, 0.007, 0.013, 0.016, 0.005
24 hours	0.015, 0.015, 0.038, 0.004
36 hours	0.020, 0.016, 0.016, 0.037, 0.001, 0.005
2 days	0.022, 0.011, 0.012, 0.020, 0.001, 0.006
3 days	0.013, 0.007, 0.006, 0.010, 0.003, 0.007
4 days	0.009, 0.005, 0.003, 0.008, 0.002, 0.011
5 days	0.010, 0.001, 0.009
10 days	0.003, 0.001, 0.002, 0.004, 0.007

When lactating dairy cows were treated in a deltamethrin plunge dip residues in the milk reached their highest levels, 0.008-0.010 mg/l, on days 1-2 after treatment (Table 8).

Meat. After Hereford beef cattle were treated with a deltamethrin pour-on formulation residues in perirenal fat were higher at day 7 than at day 3 after treatment (Table 9). At the 15 ml per 100 kg application rate, which is the label rate, the highest residue was 0.27 mg/kg 7 days after treatment. This supports the current MRL, 0.5 mg/kg (fat). Occasional deltamethrin residues were detected in the liver, kidney and muscle samples (0.003-0.010 mg/kg), but in most samples residues were not detectable (<0.002 mg/kg).

Table 8. Deltamethrin residues in milk following treatment of 6 lactating dairy cows in a plunge dip containing deltamethrin (57 mg/l) and ethion (346 mg/l) in a supervised residue trial in Australia in 1982 (Knight et al., 1982). Cows were dipped after the morning milking on day 0. Residues were measured in the rendered butterfat, and have been calculated on a whole milk basis by dividing the concentration in the butterfat by 25.

Interval after treatment, days. 1/	Deltamethrin residues in milk, mg/kg
0 M	<0.001 (6 samples)
0 E	0.004, 0.003, 0.003, 0.004, 0.005, 0.005
1 M	0.009, 0.008, 0.007, 0.006, 0.006, 0.006
1 E	0.007, 0.007, 0.006, 0.007, 0.007, 0.007
2 M	0.010, 0.008, 0.006, 0.006, 0.006, 0.007
2 E	0.006, 0.007, 0.005, 0.006, 0.006, 0.006
3 M	0.004, 0.004, 0.004, 0.004, 0.003, 0.003
4 M	0.003, 0.003, 0.003, 0.003, 0.003, 0.002
5 M	0.001, 0.002, 0.002, <0.001, 0.001, 0.002
10 M	<0.001 (6 samples)

1/ E: evening milking. M: morning milking.

Table 9. Deltamethrin residues in perirenal fat following application of an aqueous suspension pour-on formulation (7.5 g/l) directly to 15 Hereford beef cattle at 15 ml per 100 kg live-weight (1.3-1.6 mg ai/kg) and to 15others at 30 ml per 100 kg live-weight (2.8-3.2 mg ai/kg) in a supervised trial in Australia in 1991 (Wynn et al., 1991).

Post-treatment interval to slaughter, days	Deltamethrin residues in perirenal fat, mg/kg	
	15 ml/100 kg	30 ml/100 kg
1	0.077, 0.011, 0.018	0.040, 0.21, 0.16
3	0.027, 0.15, 0.17	0.26, 0.17, 0.22
7	0.27, 0.16, 0.22	0.38, 0.61, 0.33
14	0.13, 0.086, 0.17	0.29, 0.22, 0.40
28	0.043, 0.072, 0.056	0.061, 0.12, 0.13

Hereford weaner cattle (170-220 kg live-weight) were treated with a deltamethrin spot-on formulation at 2 mg ai/kg live-weight (twice the recommended rate) in a 1981 supervised trial in Australia (Roubos 1981). Deltamethrin residues in the fat samples taken at slaughter 1-14 days after treatment are shown in Table 10. The results were corrected for analytical recoveries, mean 65.3%, range 45.9-77.8%. Differences between residues in omental and perirenal fat were insubstantial, and there was little change of residue levels with time.

Table 10. Deltamethrin residues in omental and perirenal fat following application of a spot-on formulation (25 g/l xylene-based) directly to 12 Hereford weaner cattle at 4 ml per 50 kg live-weight (1.9-2.1 mg ai/kg) in a supervised trial in Australia in 1981 (Roubos. 1981).

Interval to slaughter, days	Deltamethrin residues in fat, mg/kg	
	Omental fat	Perirenal fat
1	0.049, 0.040, 0.016	0.041, 0.033, 0.017
3	0.044, 0.031, 0.026	0.051, 0.067, 0.031
7	0.040, 0.040, 0.046	0.061, 0.043, 0.034
14	0.027, 0.034, 0.046	

In a 1989 supervised New Zealand trial on calves deltamethrin was applied as a pour-on aqueous suspension formulation (Wakelin et al., 1989). The highest residue in perirenal fat was 0.27 mg/kg (Table 11). Deltamethrin residues were not detected (<0.05 mg/kg) in the liver.

Table 11. Deltamethrin residues in perirenal fat following application of a pour-on formulation (7.5 g/l micronised aqueous suspension) directly to 10 calves at 10 ml per 100 kg live-weight in a supervised trial in New Zealand in 1989 (Wakelin et al., 1989). Treatment rate was 0.75-1.1 mg ai/kg live-weight for the 5 animals slaughtered 14 days after treatment, and 1.0-1.3 mg ai/kg for the 5 slaughtered at the 21-day interval.

Post-treatment interval to slaughter, days	Deltamethrin residues (mg/kg) in perirenal fat
14	0.12, 0.27, 0.09, 0.16, 0.19
21	0.17, <0.05, <0.05, 0.09, 0.05

FATE OF RESIDUES

In storage and processing

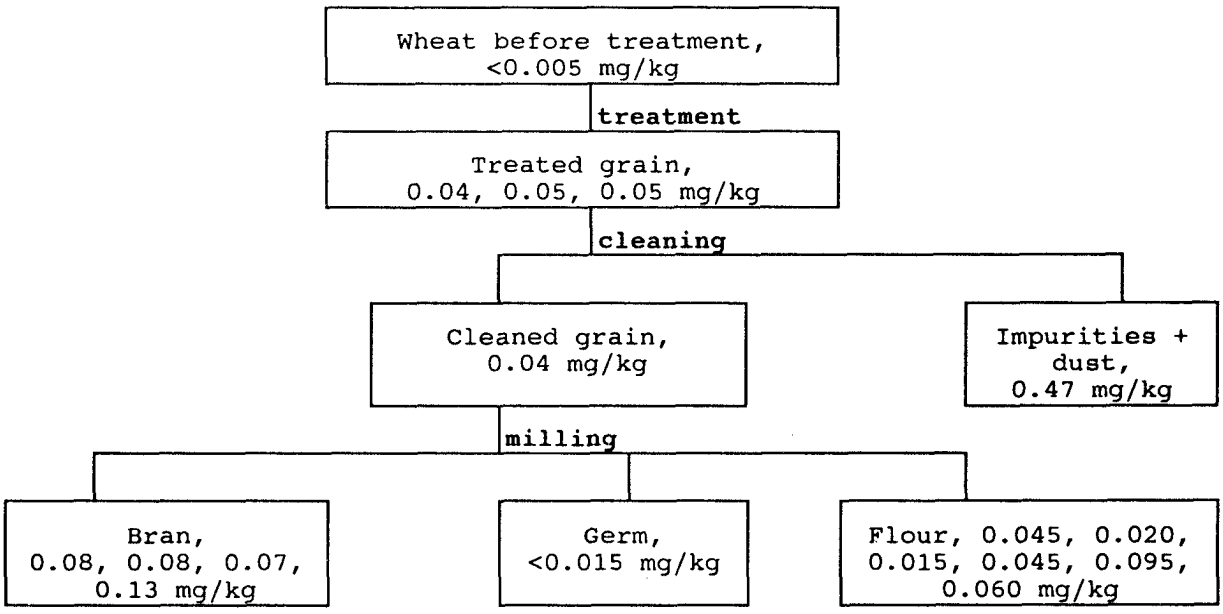
Soft wheat (250 t) was treated with deltamethrin (nominal 0.5 g ai/t) and piperonyl butoxide (nominal 4 g ai/t) in a supervised milling and baking trial in France in 1990 (Dupont, 1991). The ULV formulation contained deltamethrin (6 g/l) and technical piperonyl butoxide (60 g/l). Grain conditions were moisture 11.9%, temperature 12.3°C. One day after treatment all the wheat (250 t) was milled and samples of commodities were taken for analysis. Deltamethrin residues in the milled commodities are shown in Fig 1

In the 1991 French trial (Gervaise and Beucher, 1991b) soft wheat (216 t) was treated with a nominal deltamethrin dose of 0.5 g ai/t (calculated level after treatment 0.36 g ai/t) using a UL formulation containing deltamethrin (6 g ai/l) and technical piperonyl butoxide (60 9 ai/l). Grain conditions were moisture 13.2%, temperature 19.5°C. Deltamethrin residues in the treated grain were only about 20% of the calculated value. The remainder was eliminated during ventilation of the treated area. Five days after treatment the 216 tonnes of wheat were transported to the mill, and one day later milled. Deltamethrin residues in the milled commodities are shown in Fig 2.

The results of the milling studies revealed that residues in flour from the central part of the grain were at lower levels than in flour from the exterior of the grain. However, the grain had not been stored for any length of time so there was little opportunity for migration of deltamethrin into the grain.

Bread and cakes were produced from the flour and bran from both milling trials (Dupont, 1991; Gervaise and Beucher, 1991b). Deltamethrin residues in the baked products are shown in Table 12. The white bread mixtures consisted of 59% flour and 38% water. Wholemeal was made from a flour/bran ratio of 80/20. The Genoa cake mixture contained 28% flour, the remainder being eggs, sugar and baking powder. The croissant mixture contained 45% flour, 26% water and 25% margarine.

Fig 1. Deltamethrin residues in wheat and milled commodities from a supervised milling trial in France in 1990 (Dupont, 1991).



Supervised commercial and pilot milling trials on wheat were conducted in Australia in 1991 (Webley and Caddick, 1992). Wheat (200 tonne lots) was treated at nominal deltamethrin rates of 0.25 and 0.5 g ai/t. The formulation was an emulsifiable concentrate containing 10 g deltamethrin/l and 200 g chlorpyrifos methyl/l. Grain conditions at treatment were moisture, 9.5% and 9.2%, temperature 24.9°C and 26.5°C (0.25 and 0.5 g/t respectively). The wheat was turned upon itself for a 30 minute period 9 days after treatment and sampled for analysis to estimate the actual treatment rates. Nominal rates, calculated rates, and measured residues were 0.25, 0.26 g/t, 0.19 mg/kg and 0.5, 0.52 g/t, 0.62 mg/kg, respectively.

Fig 2. Deltamethrin residues in wheat and milled commodities from a supervised milling trial in France in 1991 (Gervaise and Beucher, 1991b).

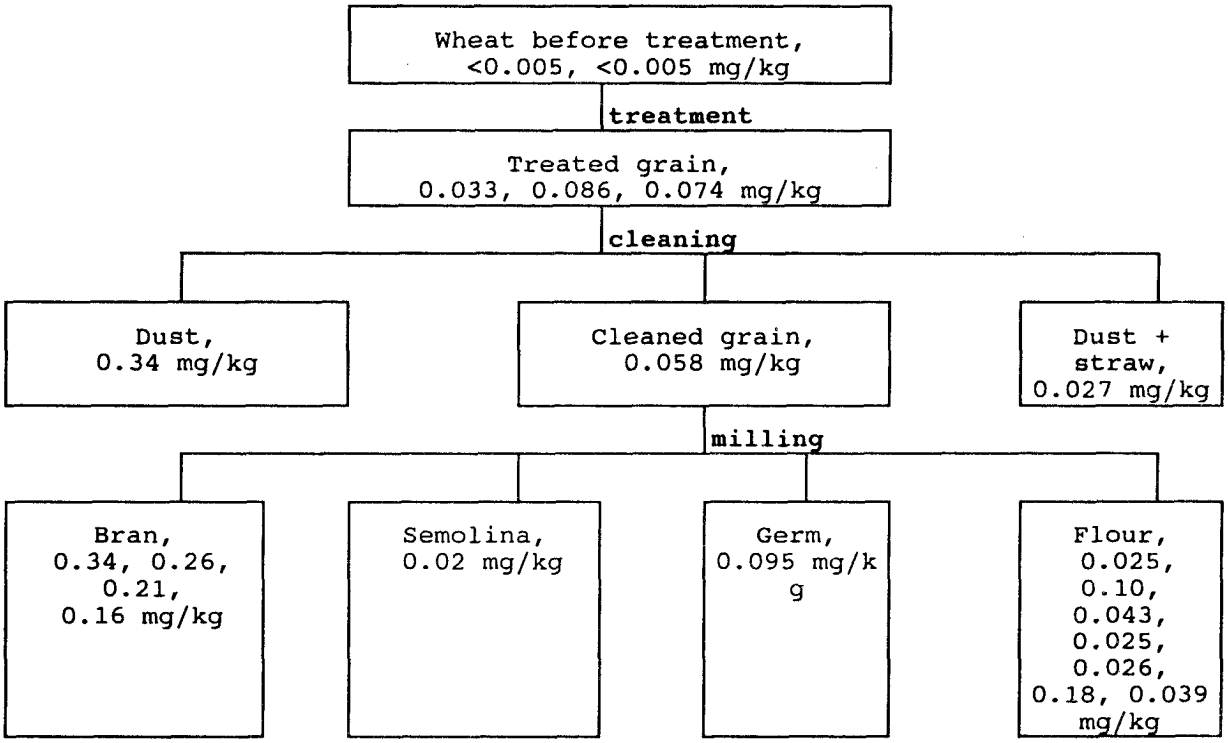


Table 12. Deltamethrin residues in bread, cake etc. prepared from milled commodities originating from deltamethrin treated wheat (Ref 3, Dupont, 1991, and Ref 5, Gervaise and Beucher, 1991b).

Commodity	Deltamethrin residues, mg/kg			
	Before baking		After baking	
	Ref 3	Ref 5	Ref 3	Ref 5
White bread, classical	0.008	0.028	<0.005	<0.005
White bread, traditional	0.006	0.027	<0.005	<0.005
Brioches	<0.015		<0.015	
Wholemeal bread	0.015	0.059	0.015	0.018
Croissants, rolls	<0.015	0.006	<0.015	<0.005
Genoa cake	<0.015	0.006	<0.015	<0.005

On two separate occasions, 8 and 22 weeks after treatment, 3 tonnes of wheat from each treatment rate were delivered to the pilot mill. The mill has been described by Orth, 1985. Deltamethrin residues in wheat and milling products are shown in Table 13.

Deltamethrin residues were measured on cooked products prepared from the pilot mill flour (Table 15). The cooked products were white bread, wholemeal bread, flat bread (Arabic style), steamed bread (Chinese style), yellow alkaline noodles and white salted noodles. Straight-run flour was used to prepare all cooked products except wholemeal bread and flat bread,

which required a dough prepared from 90:10 wholemeal flour. Commercial pan breads were produced in a pilot bakery.

Table 13. Deltamethrin residues in wheat and milling products from a supervised trial (Trial R2A) in Australia in 1991. Wheat (3 tonne lots) was processed in a pilot flour mill (throughput 660 kg wheat/hour). Two samples (wheat and each milled commodity taken at different times during the milling process) were both analysed by two laboratories (Webley and Caddick, 1992; Shields, 1991).

Treatment	Deltamethrin residues, mg/kg							
	0.25 g ai/ t				0.5 g ai/t			
Wheat storage time	8 weeks		18 weeks		8 weeks		18 weeks	
Laboratory 1/	AGT	AC	AGT	AC	AGT	AC	AGT	AC
Wheat 2/	0.2, 0.2	0.16, 0.11	0.14, 0.13	0.20, 0.18	0.45	0.39, 0.34	0.31, 0.36	0.41, 0.34
Bran	0.7, 0.6	0.60, 0.58	0.60, 0.55	0.78, 0.69	1.8, 1.8	1,6, 1.4	1.5, 1.5	1.4, 1.5
Germ	0.2, 0.2	0.29, 0.34	<0.02, 0.06	0.28, 030	0.6, 0.6	0.60, 0.66	0.58, 0.60	0.56, 039
Flour, straight run	< 0.02	0.10, 0.11	0.02 0.04	0.06, 0.06	0.1, 0.1	0.30, 031	0.12, 0.12	0.15, 0.09
Flour, last reduction (contains some bran)	0.09, 0.10	0.04, 0.04	0.05, <0.02	0.14, 0.14	0.27, 034	0.12, 0.10	0.30, 0.31	0.36, 0.32
Wholemeal 90: 10			0.03	0.21			< 0.02	0.37

1/ AGT: Academy of Grain Technology. AC: Analchem.
2/ Cleaned wheat, to first break, sampled during the milling run

Wheat was delivered to two commercial mills for processing, 50 tonnes of each treatment to Weston Milling, and 25 tonnes to Bunge Albury Mills (Webley and Caddick, 1992). Residues in the various fractions are shown in Table 14. Reconstituted wheatmeal consisted of flour and middlings 80%, and bran and pollard 20%.

Table 14. Deltamethrin residues in wheat and milling products from a supervised trial (Trial R3A) in Australia in 1991. Wheat (50 and 25 tonne lots) was processed in two commercial mills. Two samples (wheat and each milled commodity taken at different times during the milling process) were both analysed by two laboratories. (Webley and Caddick, 1992; Shields, 1991).

Treatment	Deltamethrin residues, mg/kg							
	0.25 g ai/ t				0.5 g ai/t			
Wheat storage time	10 weeks		15 weeks		10 weeks		15 weeks	
mill	Weston Milling Co		Bunge Albury		Weston Milling Co		Bunge Albury	
Laboratory 11	AGT	AC	AGT	AC	AGT	AC	AGT	AC
Wheat, after cleaning	0.11, 0.11	0.16, 0.20	0.08, 0.08	0.21, 0.18	0.26, 0.25	0.41, 0.42	0.31, 0.32	0.39, 0.45
Bran	0.38, 033	0.49, 0.46	0.29, 0.25	0.69, 039	0.70, 0.43	1.2, 1.0	0.91, 1.0	1.2, 1.4
Germ	0.10, 0.11	0.15, 0.18	<0.02	0.15, 0.18		0.39, 0.29	0.27	0.33, 0.39
Flour, straight run	0.02	0.09, 0.20	<0.02	0.07, 0.10	0.02	0.15, 0.10	0.15	0.15, 0.16
Flour, last reduction	0.02	0,05, 0.06	<0.02	-0.15,0.15	0.10, 0.13	0.22, 0.15		0.29, 026
Wheat meal				0.11				0.32

1/ AGT: Academy of Grain Technology AC: Analchem

Table 15. Deltamethrin residues in cooked products prepared from milled commodities originating from deltamethrin treated stored wheat (Webley and Caddick, 1992). Flour and wholemeal were generated in Trial R2A (Table 13).

Treatment	Deltamethrin residues, mg/kg			
	0.25 g ai/t		0.5 g ai/t	
Wheat storage time	8 weeks	18 weeks	8 weeks	18 weeks
Flour	0.04	0.02	0.10	0.11
Wholemeal 90: 10	0.10	0.15	0.42	0.41
White bread	0,03, 0.03	0.02, 0.02	0.06, 0.06	0.04, 0.03
Wholemeal bread	0.07, 0.07	0.04, 0.02	0.26, 0.22	0.15, 0.15
Flat bread	0.08, 0.07	0.06, 0.07	0.20, 0.20	0.20, 0.20
Steamed bread	0.02, 0.02	0.02, 0.02	0.06, 0.05	0.06, 0.05
Yellow alkaline noodles	0.02, 0.02	0.02, 0.02	0.09, 0.09	0.07, 0.06
White salted noodles	0.02, 0.02	0.02, 0.02	0.06, 0.05	0.05, 0.03

Table 16. Deltamethrin residues in treated wheat in a series of trials in Italy in 1985-86 (Molinari, 1987).

Site	Applic rate, g ai/t		Deltamethrin residues (mg/kg) on wheat after treatment, Analyses on replicate samples
	Nominal	Calc	
LENDINARI, soft	0.25	0.15	<0.01, 0.021, 0.052
wheat (80 t), vertical silo	0.5	0.31	0.01, 0.021, 0.060
	1	0.96	0.21, 0.17, 0.13
LENDINARI, soft	0.25		0.10,0.08
wheat (30 t), horizontal store	0.5		0.061, 0.18, 0.088
	1		0.25, 0.17, 0.21
PONTE A RIGO, soft wheat	0.25		0.085, 0.13, 0.11
(400 t),horizontal store	0.5		0.32, 0.35, 0.37
	1		0.46, 0.45, 0.41
LA SPEZIA, hard	0.25	0.19	0.021, 0.093, 0.17
wheat (3900 t), vertical silo	0.5	0.44	0.066, 0.038, 0.014
	1	1.03	0.44, 0.20, 0.26
SAN GIORGIO DI PIANO,	0.25	0.23	<0.01, 0.15, 0.052, 0.16, 0.033
hard wheat (900 t), vertical silo	0.5	0.5	0.021, 013, 0.055, 0.26, 0.13
	1	1.1	0.083, 0.24, 0.081, 0.21, 0.24
MONTE PESCALI, hard	0.25		0.13, 0.15, 0.097
wheat (540t), vertical silo	0.5		0.25, 0.20, 0.27
	1		0.34,0.23
TURREME, hard	1	1	0.22, 0.42, 0.76
wheat (3 t), horizontal store	2	2	1.3, 13, 1,2

The extensive set of trials on stored wheat conducted by Molinari (1987) has already been reviewed by the 1988 JMPR. However, the replicate analyses on the wheat provide valuable information on the precision of sampling and analysis of such residues (Table 16). The data suggest caution in interpreting small relative differences in concentrations, particularly at lower levels.

Fig 3. Piperonyl butoxide residues in wheat and milled commodities from a supervised milling trial in France in 1990 (Dupont, 1991).

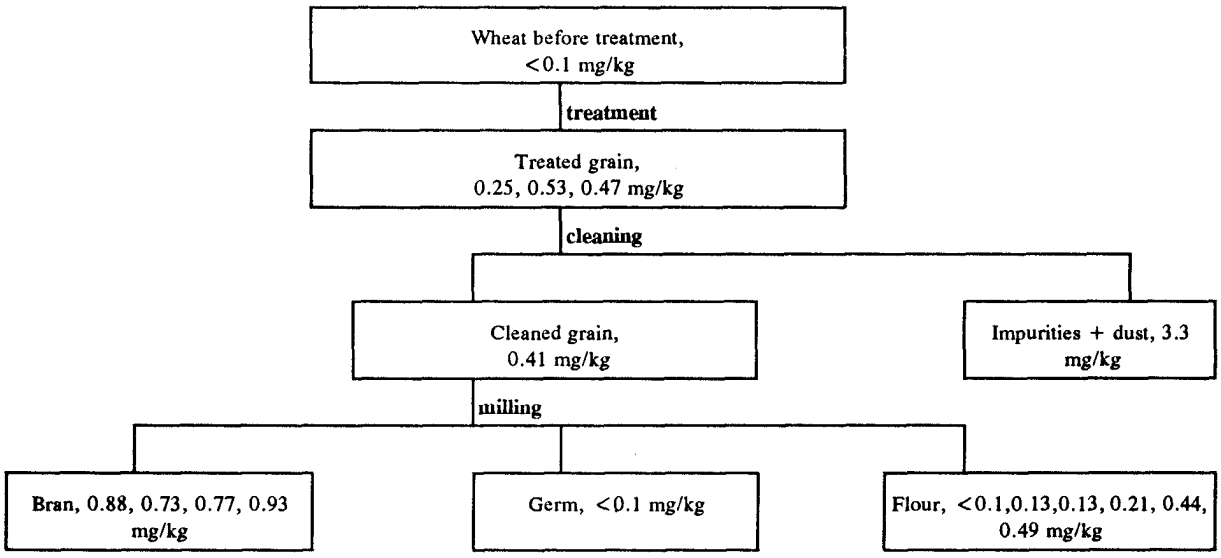


Table 17. Piperonyl butoxide residues in bread, cake etc. prepared from milled commodities originating from piperonyl butoxide-treated wheat (Dupont, 1991).

Commodity	Piperonyl butoxide residues, mg/kg	
	Before baking	After baking
White bread, classical	0.15	<0.1
White bread, traditional	<0.1	<0.1
Brioches	<0.1	<0.1
Wholemeal bread	0.30	0.13
Croissants, rolls	<0.1	<0.1
Genoa cake	<0.1	<0.1

The fate of piperonyl butoxide was investigated during milling and baking at the same time as the deltamethrin trials (Dupont, 1991). In the same way as with deltamethrin, residues are depleted during the cleaning process, are more concentrated in the bran and less in the flour (Fig 3). The cooking studies (Table 17) provide little information because residues were mostly undetectable in the mixtures before baking, but in wholemeal bread and classical white bread residues were depleted in the cooked product.

Residues in the edible portion of food commodities

When deltamethrin is used commercially as a grain protectant likely residues in cooked products would be white bread <0.005-0.05 mg/kg, wholemeal bread and flat bread 0.02-0.2 mg/kg, noodles 0.02-0.1 mg/kg.

METHODS OF RESIDUE ANALYSIS

In the French cereal processing studies wheat and its commodities were analysed for deltamethrin residues by a GLC method (Gervaise and Beucher, 1991a). The extracting solvent was acetonitrile. The method used cyhalothrin as an internal standard and had a limit of determination of 0.005 mg/kg.

In the Australian studies on wheat commodities samples were also analysed by GLC. The Academy of Grain Technology laboratory used AWB Method P15 (Swinden, 1989). The extracting solvent was toluene. The limit of determination was 0.02 mg/kg. Analchem Consultants laboratory used method PR33, which is very similar to method P15.

In the Australian milk trials butterfat was dissolved in hot hexane, and the deltamethrin residues were then partitioned into acetonitrile. After gel-phase chromatography clean-up deltamethrin was measured by GLC. The method was based on Wellcome Foundation Ltd method HIBH 82-1. The same method was used for the analysis of beef fat.

An analytical method (Nicolas, 1985) previously reviewed in 1990 was used in the French dairy cow studies (de Laistre Banting, 1989a,b).

NATIONAL MAXIMUM RESIDUE LIMITS

The Meeting was aware that the following MRLs for deltamethrin had been established.

Country	MRL, mg/kg	Commodity
Australia	5	wheat bran
	2	cereal grains (whole grain)
	0.2	milk (fat basis)
	0.1	fat of meat of cattle, goats and sheep
France	3	bran
	2	green coffee beans
	1	grain legumes, wheat
	0.2	white flour (proposed)
Italy	1	cereals
Morocco	0.5	cereals
South Africa	1	oats, rye, wheat
Spain	1	cereal grains, pulses
	0.5	leafy vegetables
	0.2	vegetables (except as otherwise listed)
	0.05	citrus fruits, grapes, olives, pome fruits, stone fruits
	0.01	tuber, root and bulb vegetables, lucerne

APPRAISAL

Deltamethrin is registered in at least 15 countries for the control of stored product insects in cereals, at 0.5-1 g ai/t when used alone or at 0.25-0.5 g ai/t when synergised by piperonyl butoxide. Information had been previously provided on the fate of deltamethrin residues on wheat during milling and baking, but it was suggested at the 20th (1988) Session of the

CCPR that the proposed MRL (2 mg/kg) for unprocessed wheat bran was too low. The proposed MRLs for wheat bran, wheat flour and wheat wholemeal were held at Step 7B pending review of data to be supplied by the manufacturer.

Information on milling and baking studies on deltamethrin-treated wheat in France in 1990 and 1991 was made available to the Meeting. Supervised commercial and pilot milling trials and cooking studies from Australia were also provided.

Residues in the milled and cooked commodities depend first on the level of residues in the wheat. The level applied, established by grain analysis soon after application, can vary from approximately 10 to 100% of the nominal value. Such values are recorded in the data provided to the present Meeting, which substantially agree with the range 15-85% recorded in the 1980 and 1982 Evaluations. Efficiency of application can be low with a UL formulation applied directly to the grain stream at 80 ml of UL formulation (6 g ai/l) per tonne of grain. Much of the deltamethrin is attached to dust which separates from the grain during treatment.

Because much of the residue is in the dust or particulate matter, residues may also be lost during transport (some separation of dust from grain) and during the cleaning process, which is the first step in commercial milling. Possibly 10-20% of the residue is removed during the cleaning.

From information supplied to the present Meeting, the level of deltamethrin residues in bran was 2.3-4.7 times the level in the cleaned wheat (mean 3.5 times). The mean was 3.6 (residues in bran + residues in wheat) in trials recorded in the 1988 Evaluations.

Residues in the bran depend on the treatment rate, efficiency of application, percentage of residues remaining after cleaning, and ratio of residues in the bran to residues in the wheat. The maximum, most likely and minimum for these four quantities are [1, 1, 0.9, 4.7], [0.5, 0.5, 0.85, 3.5] and [0.25, 0.1, 0.8, 2.3] respectively. The maximum, most likely and minimum residues in bran then become 4.2, 0.74, and 0.05 mg/kg respectively. The figures suggest a maximum residue level of 5 mg/kg for bran, but also suggest that when deltamethrin is used at a lower treatment rate at a typical application efficiency residues in bran are likely to be under 1 mg/kg.

Similar logic applies to residues in flour, although there is an additional complication with the different streams of flour from the mill. Because the residue is associated with the outer portion of the grain those streams containing a higher proportion of the outer grain are likely to have higher residues. Data from Australian milling trials suggest that 0.2 mg/kg would be a suitable maximum residue level for straight-run flour, but residues in last-reduction flour exceeded 0.2 mg/kg. Again, typical residues in flour are likely to be rather lower if deltamethrin is applied at typical application efficiencies. Generally, residues in flour milled from deltamethrin-treated wheat would be less than 0.1 mg/kg.

Data supplied to the Meeting and to the 1981 and 1988 Meetings show that residues in wholemeal are likely to be 70-100% of residues in the wheat.

The 1981 JMPR drew some conclusions about the effect of baking on deltamethrin residues. Essentially, there is no deltamethrin degradation during baking, but there is some dilution because of the higher moisture content of the bread. Data provided to this Meeting would not change that conclusion.

When deltamethrin is used commercially as a grain protectant likely residues in cooked products would be white bread <0.005-0.05 mg/kg, wholemeal bread and flat bread 0.02-0.2 mg/kg, noodles 0.02-0.1 mg/kg.

Supervised trials in New Zealand demonstrated that when deltamethrin was used on tamarillos (tree tomatoes) according to GAP, 0.6-1.25 g ai/hl and 60 days PHI, residues were close to 0.01 mg/kg.

Residue data from Spain showed that, at an application rate of 0.26 kg ai/ha (a greatly exaggerated rate) on artichokes, deltamethrin residues were within the CXL for globe artichoke (0.05 mg/kg) 7 days after application.

At the 24th (1992) Session of the CCPR comments were made (ALINORM 93/24, para 140) about the MRL for meat, particularly with respect to the waiting period before slaughter after veterinary treatments. Data from Australia and France were provided to the Meeting on residues in milk and meat after treatment of cattle with deltamethrin. Residues in milk reached a peak from day 1 to day 3 after treatment, with wide variations between levels in milk from individual animals. Data on deltamethrin residues in fat from treated animals suggest that residues at day 7 after treatment are either the same as or slightly higher than at day 3. Observing 3 or 7 days withholding periods for pour-on treatments would not reduce deltamethrin residues at slaughter.

The Meeting confirmed the previous MRL recommendations for unprocessed wheat bran (5 mg/kg) and wheat wholemeal (1 mg/kg).

RECOMMENDATIONS

on the basis of the data on residues from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits.

Definition of the residue: deltamethrin (fat-soluble).

Commodity		MRL, mg/kg	Previous MRL, mg/kg
CCN	Name		
FT 0312	Tree tomato	0.02	
CF 1211	Wheat flour	0.2 PoP	0.1 Pop

REFERENCES

1. de Laistre Banting, A. 1989a. Determination of residues in milk of deltamethrin after external application of "Butox pour on" to dairy cows. Cebiphar. Report E.340. Unpublished.

2. de Laistre Banting, A. 1989b. Determination of residues in milk of deltamethrin after external application of Buttox "Pour On" to dairy cows. Cebiphar. Report E.315. Unpublished.

3. Dupont, P.L. 1991. Report on pesticide residue trial, K-OBIOL ULV 6PB on wheat. Roussel Uclaf. Unpublished.

4. Gervaise, Y. and Beucher, I. 1991a. Mise au point du dosage de residus de deltamethrine. Crépin Analyses et Contrô1es. Unpublished.

5. Gervaise, Y. and Beucher, I. 1991b. Étude de dosage des residus de deltamethrine dans les échantillons de blé et dérivés. Crépin Analyses et Contr61es. Unpublished.

6. Kieran, P.J., Parker, R.C., Prested, C. and Winter, R.L. 1981. Determination of residues of NRDC 161 in rendered butterfat derived from cows treated with a xylene-based NRDC 161 pour-on formulation. Wellcome Group Research and Development. Document HITH 81/1. Unpublished.

7. Knight, P.J., Parker, R.C., Prested, C. and Tucker, C.M. 1982. Determination of residues of deltamethrin in rendered butterfat derived from cattle dipped in a mixture of deltamethrin and ethion. Wellcome Group Research and Development. Document HIBH 82-3. Unpublished.

8. Molinari, G.P. 1987. Use of deltamethrin in grains storage: efficacy and residue assessment in the treated products and in the derived foodstuffs. Roussel Hoechst Agrovet. Unpublished.

9. Nicolas, P. 1985. Determination of deltamethrin in milk. Cebiphar. Technique CPG.100.11.85. Unpublished.

10. Roubos, H. 1981. A report on a residue study in cattle with a deltamethrin 25 g/1 low volume spot-on formulation. Wellcome Group Research and Development. Document AITH 81/2. Unpublished.

11. Orth, R.A. 1985. Pilot flour mill for Australia. Food Technology in Australia. 37, 318-319.

12. Shepherd, S., Wynn, B.L., O'Neill, P. and Garrod, J. 1991. A report detailing a milk residue study in dairy cattle treated with a deltamethrin pour-on lousicide. Pitman-Moore. Document AIPN 91-1. Unpublished.

13. Shields, R. 1991. Determination of deltamethrin in wheat and milled products. Analchem ref 91/1515, 91/1602, 91/1880, 91/2358. Unpublished.

14. Swinden, L. 1989. Determination of deltamethrin residues in wheat, wheat fractions and bread using gas liquid chromatography. Method no. P15. Academy of Grain Technology, Australia. Unpublished.

15. Wakelin, R.L., Collins, K.R. and Shakes, T.R. 1989. A research report on the residues of deltamethrin in fat and liver samples from 3-5 month old calves following the administration of an aqueous backline pour-on preparation containing 0.75% deltamethrin. Pitman-Moore. Reference BU 89-104. Unpublished.

16. Webley, D. and Caddick, L. 1992. Deltamethrin residues on wheat, milled and cooked products following commercial treatment, commercial milling, pilot milling and baking. Australian Wheat Board and the Flour Millers' Council. Report AGT/92/R/1. Unpublished.

17. Wynn, B.L., Shepherd, S. and O'Neill, P. 1991. A report detailing a tissue residue study in beef cattle treated with a deltamethrin pour-on lousicide. Pitman-Moore. Document AIPH 91-2. Unpublished.